Detailed Study of Contaminant Production in a Space Cabin Simulator at 258 mm. Mercury

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Additional information has been accumulated concerning the contaminants associated with habitation by man of a closed confined space. An experiment designed to determine man's contribution to trace contaminants was conducted jointly by the United States Air Force and the National Aeronautics and Space Administration. The experiment was divided into three phases: an unmanned period, a manned period, and a manned period coupled with an activated carbon scrubber. Direct analyses of the sealed environment were not adequate for this comprehensive survey; however, cryogenic fractionation and concentration provided samples with sufficient concentration of contaminants for analysis by means of gas chromatography, infrared spectroscopy, and mass spectroscopy. Of the 142 compounds identified and quantified during the experiment, only 45 were found during the manned phases.

THE DEVELOPMENT AND USE of analytical procedures to detect trace atmospheric constituents, coupled with experiments specifically designed for the appraisal of man as a production or removal system, or both, will serve to define further the problems associated with man's subsistence in closed-loop confined spaces. These endeavors are of value for ascertaining physiologic as well as instrumental requirements placed upon life support systems to maintain human existence without incurring debilitating conditions that might jeopardize mission accomplishment.

Extensive reporting of material off-gassing products has been combined with computer retrieval systems to make available data on the contributions that could be made from materials to the atmosphere.[2-10] Detailed reports have been published which elucidate the matrix generated by man and his environment in submarines, spacecraft, and ground-based simulators. Likewise, an earlier experiment specifically designed for trace contaminant study provided information concerning man's contribution to the atmosphere.[1]

The purpose of this paper is to present the results of a joint study conducted by the United States Air Force and the National Aeronautics and Space Administration designed to define within the limits of the analytical procedures[6,9,12] those contaminants resulting from human habitation of a sealed environment in an oxygen atmosphere at 258 mm. Hg total pressure.

METHODS

An experiment was conducted in a test cell[1] maintained at a total pressure of 258 mm. Hg. The study was divided into three phases: an 8-day unmanned background period, a 14-day manned period, and a 7-day manned period with an activated carbon scrubber. The initial phase established the atmospheric matrix resulting from the test cell supply gases, and previous occupancy by man. The second phase provided information as to the change in the atmospheric matrix by the inclusion of man into the system. The final phase demonstrated the effect of an activated carbon scrubber on the man-influenced atmospheric matrix. The gross atmospheric composition during the three phases is presented in Table I.

Experimental Subjects—Four male Air Force volunteer subjects, ages 19-20 years, participated in the study. All of the subjects were in good physical condition, hav-

TABLE I. GROSS ATMOSPHERIC COMPOSITION

Major Atmospheric Components		Unmanned	Manned	Manned-plus-Charcoal
Oxygen	Maximum	98.8%	96.2%	92.0%
	Minimum	96.0%	90.0%	89.9%
	Mean	96.7%	92.7%	91.0%
CO_2	Maximum	0.97%	1.62%	1.47%
	Minimum	0.0%	0.0%	0.15%
	Mean	0.07%	0.76%	0.69%
Nitrogen	Maximum	2.8%	6.35%	6.90%
	Minimum	0.35%	2.4%	4.35%
	Mean	1.96%	4.74%	6.47%
Total pressure mean		256 mm. Hg	256.7 mm. Hg	257.8 mm. Hg

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ing recently completed basic training. As in the previous study,[1] only individuals who produced methane in their flatus were selected. The flatus of the subjects was collected after the manner of Kirk[6] and analyzed by gas chromatography. The physical characteristics of these individuals are presented in Table II. The subjects underwent complete physical examinations prior to entering the test cell and after completing the 21-day experimental phase. The physical examination included ophthalmologic, ear-nose-throat, neurologic, and psychiatric evaluations. In addition, standard chest, abdominal, and paranasal sinus roentgenographs, electrocardiograms, and electroencephalograms were obtained. Throughout the entire 21-day manned phase and for four days pre- and post-experimentally, the subjects were maintained on a total liquid diet,* ad libitum water intake, and a daily vitamin-mineral supplement** with an additional 26 mEq of potassium chloride in the enteric coated form. The 24-hour intake was adequate in all necessary dietary constituents.

The subjects were taught to obtain vital signs and to perform venipunctures on one another. Vital signs were measured daily and venipunctures were performed three times weekly. Blood, urine and feces samples were sent to the outside through a small passlock without altering the environmental conditions. A complete medical review of systems was performed by a physician at least once daily through an intercommunication system.

Test Cell Operation—The test cell was provisioned with food and other logistical support items before the unmanned background portion of the experiment was initiated. During the 21-day manned portion of the test, the fecal material was stored in plastic bags in a freezer before being removed from the test cell. Urine which was not retained for analysis was removed via a water trap from the test cell. The trap was well flushed with water following this procedure. The subjects did not shave and their hygienic activity was limited to dry brushing of the teeth and bathing with a damp cloth which was dried and stored in the test cell. Canisters containing approximately 14 kilograms of lithium hydroxide were used for carbon dioxide removal. These canisters were changed on an average every three days. The test cell had a double-walled shell, and a special control circuit was used to maintain the pressure of the annulus at the same pressure as the interior of the cell. This mode of operation provided a diffusion barrier between the ambient atmosphere and the test cell which prevented the loss of gas from the test cell and contamination from the ambient atmosphere. The portion of the test cell used during the study had a bound volume of 27.45 m.[3]

The background portion of the study was initiated by flushing the test cell with oxygen from a liquid source while the cell was being evacuated to 258 mm. Hg. This phase of the test was continued for 185.5 hours. The manned portion of the test was begun immediately following the background phase. The subjects entered the test cell through the airlock which was flushed with oxygen from a liquid source as it was reduced to the pressure of the test cell. The manned portion of the experiment continued for 503 hours. The final 172 hours of the study had a 454-gram bed of activated carbon (used without pretreatment from delivery containers) in operation with an average air flow through the bed of 0.07 m³/min.

Gas Analyses—A dual-flame gas chromatograph with two 3.05 meters by 3.2 millimeter columns packed with 4% Carbowax 4000 and 6% polyphenyl ether on 60-80 mesh Teflon was used to monitor the atmosphere of the test cell for contaminants in concentrations in excess of 10 p.p.m. Test-cell gas samples were introduced on a batch basis into the chromatograph from a sample loop which was continuously flushed with the test-cell gas. Methane concentrations were determined with a flame ionization gas chromatograph by analysis of batch samples obtained on a periodic basis. Carbon monoxide was evaluated using a Beckman IR-7 with a 10-meter multipath cell and a "Lira" 300 analyzer sensitized for carbon monoxide. Hydrogen was monitored with a Bendix 17-210 Time-of-Flight mass spectrometer. The techniques used were similar to those used in a previous study.[1]

Conventional techniques of analysis, such as gas chromatography, and infrared and mass spectrometry of unconcentrated samples, would have revealed only 5 to 10 of the 142 trace constituents reported; therefore, cryogenic trapping was utilized as the best and most practical method of obtaining concentrated samples for subsequent analysis. As in the previous study,[1] multistage cryogenic trapping systems were operated 20 to 22 hours per day. The samples were distributed to three analytical laboratories for detailed chemical analysis. The analyses were provided by the Von Karman Center of Aerojet-General Corporation,[9] Arnold Engineering and Development Center,[3] and Melpar, Inc.[12]

The average concentration of a compound in the chamber during a cryogenic trapping period was estimated from the total milligrams of the compound contained in the cryogenically obtained sample, the mass flow rate of the sample gas through the system, and the total trapping time. The total flow through the system in each trapping period was obtained by multiplying the average mass flow rate through the system by the time of the trapping period in minutes. The mass flowmeters used in the determination had an accuracy of one percent. The concentration in the test cell of the contaminant in mg/m³ was determined at 21.1°C. and 760 mm. Hg by the following equation:

*Prepared by U. S. Army Natick Laboratories, Natick, Mass.

**ABDEC with Minerals, Parke-Davis and Company, Detroit, Michigan.

TABLE II. PHYSICAL CHARACTERISTICS OF SUBJECTS

Subject Number	Age (years)	Height (cm)	Weight (kg)
90	19	184	76.7
91	20	170	68.0
92	19	188	79.4
93	20	176	77.1

$$\frac{\text{contaminant weight in milligrams}}{V} \times 10^3 = \text{milligrams/meter}^3$$

Where,

$\frac{\text{contaminant weight in milligrams}}{V}$ = the amount of material in a sample as reported by an analysis group.

V = volume of gas passed through the multistage cryogenic trapping system determined on a mass flow basis at 760 mm Hg and 21.1°C.

The observed concentration of a compound in mg/m³ was corrected by a computer program for the amount of each compound removed from the test cell and the addition of compounds from the oxygen supply.

Since the volume used in the calculations was measured with a mass flowmeter calibrated to read standard cc./minute at 760 mm. Hg rather than at 258 mm. Hg, the volume processed at the test-cell conditions was 2.95 (760/258) times greater. The concentrations in the inspired volume must be considered for all compounds (Figure 1) which are reported on a mass flow-determined volume at 760 mm Hg and 21.1°C. Man's inspired volume at 258 mm. Hg is about the same as that at ground level;[11] therefore, in order to assess the physiological significance at 258 mm. Hg, the reported concentrations must be multiplied by 0.359.

Clinical Analyses—Numerous venous blood and urine studies were performed throughout the experimental phase and for at least one week before and after the ex-

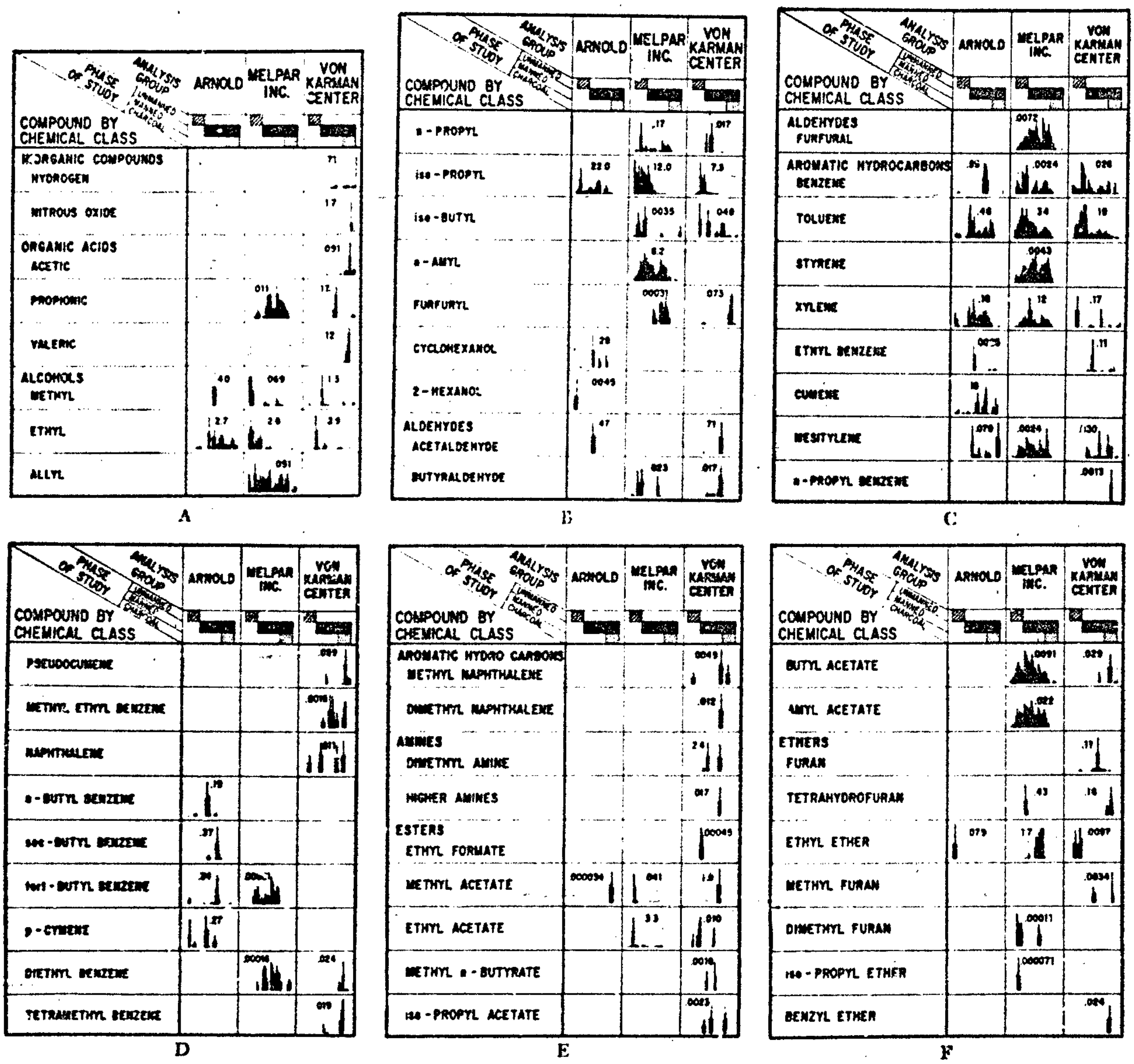
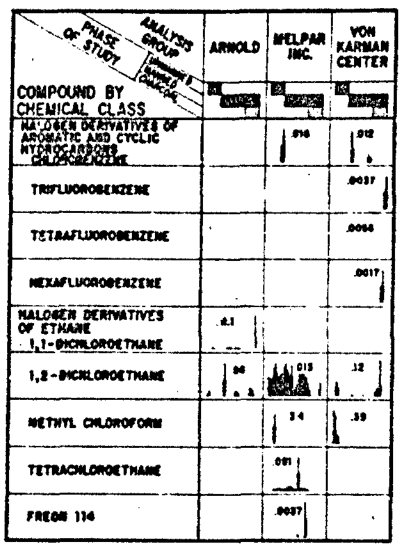


Fig. 1. Histogram of observed compounds. (For explanation see text.)

periment. Blood studies were performed three times weekly. Freshly voided morning urine specimens were analyzed for sugar, protein, acetone, specific gravity, and pH by standard laboratory methods. A careful microscopic examination was performed on the sediment. All stool samples were tested for occult blood with guaiac reagent. Complete blood counts, including hemoglobin and hematocrit determination, reticulocyte counts, white blood cell counts, and differential counts were performed by standard techniques. Serum and urine creatinines, fasting blood glucose, blood urea nitrogen, and serum alkaline phosphatase were determined by AutoAnalyzer techniques. Serum protein electrophoresis was performed with the Spinco Model R paper electrophoresis system and total serum proteins were measured by a modified biuret method.[14] Serum glutamic-oxaloacetic transaminase (SGOT) and serum glutamic-pyruvic transaminase (SGPT) were ascertained by the Sigma-Frankel procedure[13] and serum bilirubin by the method of Malloy and Evelyn.[6] Bromsulphalein retention was determined before and after the experiment utilizing standard dye injection techniques with a 45-minute venous blood measurement.

RESULTS

Atmospheric Gas Analyses—One hundred and forty-two (142) different compounds (Figures 1 and 2) were reported by the analytical groups during the experiment. The data (Figure 1) are presented in mg/m³ in the test cell at 21.1°C. and 760 mm. Hg corrected for the amount of a compound added to and removed from the test cell during the experiment. The greatest concentration, represented by the highest peak, appears at the top of each histogram with the base being equiva-



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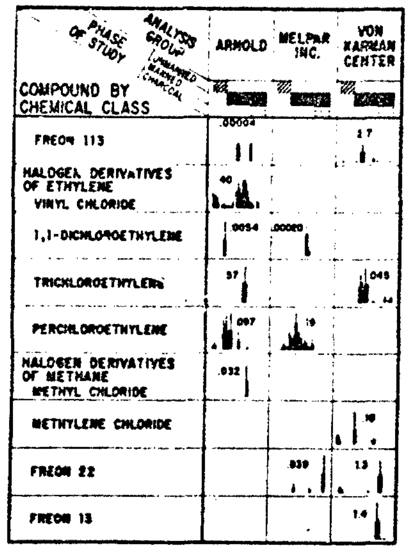

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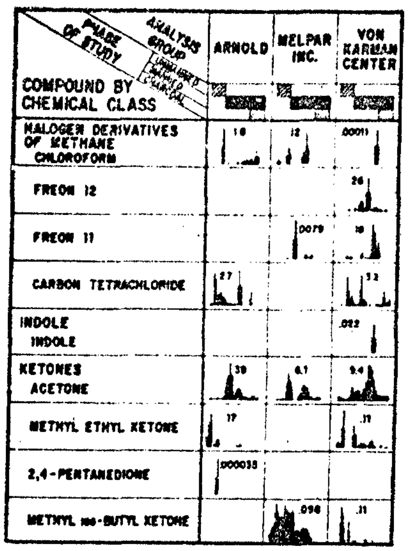

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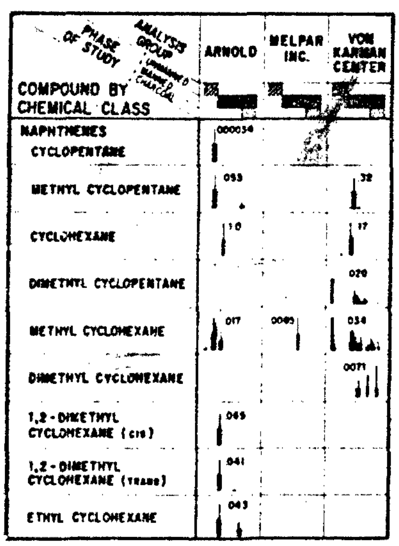

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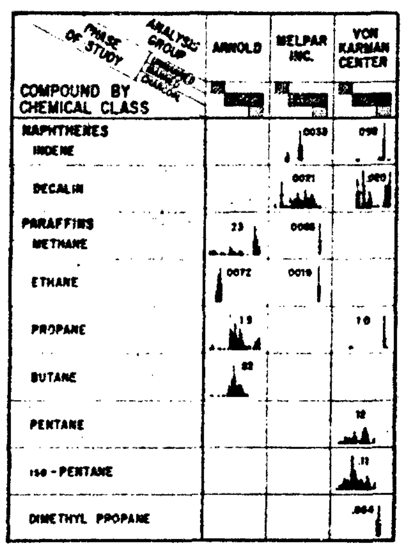

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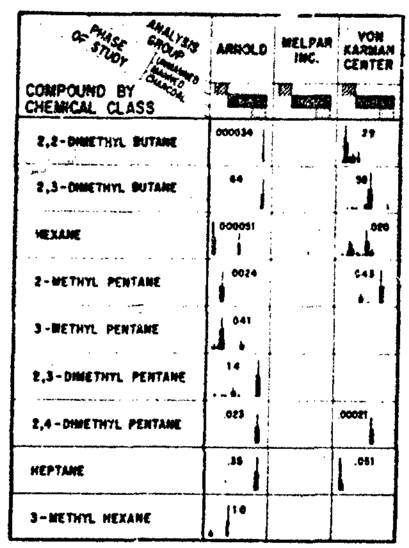

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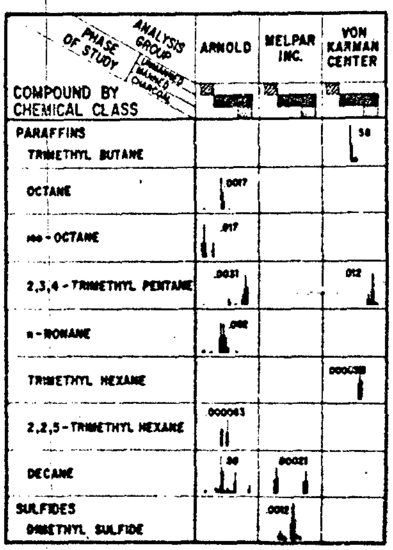
lent to zero. Time is represented on the horizontal portion of the histogram with the unmanned phase ending at 185.5 hours, the manned phase at 516.5 hours, and the manned-plus-charcoal phase at 688.5 hours. The frequency of occurrence of each compound is approximate since an interpolation between data points was necessary in the computer program used for concentration corrections. Additionally, the corrected values in Figure 1 are approximately 10% greater than the observed values.

Arnold Engineering and Development Center detected 73 compounds during the experiment of which all but carbon dioxide are shown in Figure 1. Six compounds were reported only during the unmanned phase, and 16 compounds during the manned or manned-plus-charcoal phases. The other 50 compounds were reported both in the unmanned phase and in one or both of the manned phases.



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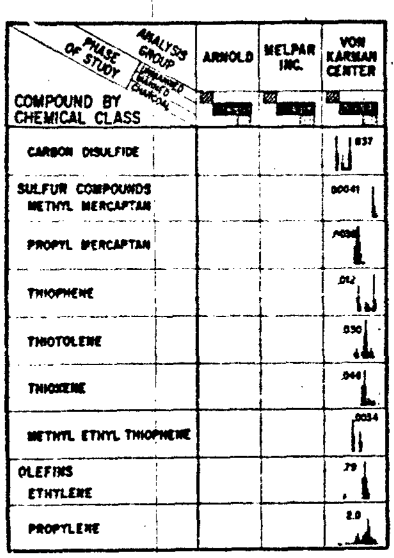
Melpar, Inc. detected 48 compounds during the experiment of which all but carbon dioxide and ethyl benzene are shown in Figure 1. Ethyl benzene is not included here because its quantities, appearing in the manned-plus-charcoal phase only, were insufficient to be handled by the computer program. Two compounds were reported only during the unmanned phase, and 10 compounds during the manned or manned-plus-charcoal phases. The remaining 34 compounds were reported in both the unmanned phase, and in one or both of the manned phases.

Von Karman Center of Aerojet-General Corporation reported carbon dioxide and the 92 compounds presented in Figure 1. Two compounds were detected only during the unmanned phase, and 51 compounds during the manned or manned-plus-charcoal phases. The other 39 compounds were reported in both the unmanned phase and one or both of the manned phases.

Clinical Observations—The four experimental subjects tolerated the environmental phase without difficulty. Complaints were minimal and most were centered on the diet and relative boredom. Subject 93 experienced mild aches in the knee joints during the initial 12 hours, which disappeared with no further complications. His symptoms were interpreted as a transient, grade 1, decompression disturbance. Subjects 90 and 93 had minimal discomfort from pressure changes in the middle ear. The need for clearing their ears was manifested primarily during the sleeping hours. No further subjective symptoms were elicited during the experiment.

Subjects 90 and 93 exhibited abnormal urinary sediments on the fourth post-experimental day. The sediment from subject 90 showed numerous coarsely granular casts (2-4 per low-power field) and occasional hyaline casts. There was no proteinuria and only an occasional white blood cell. These abnormalities cleared completely over the next four days. The sediment from subject 93 exhibited numerous red blood cells (75-100 rbc per high power field) without casts, proteinuria, or



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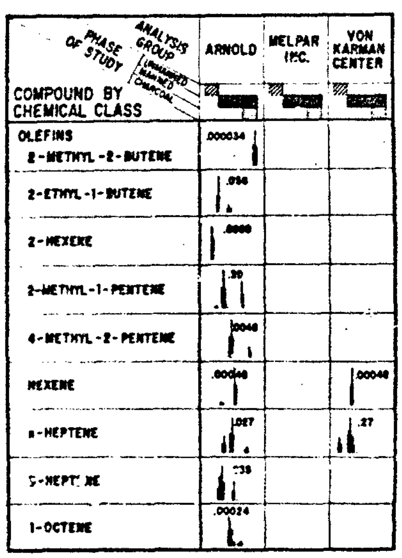

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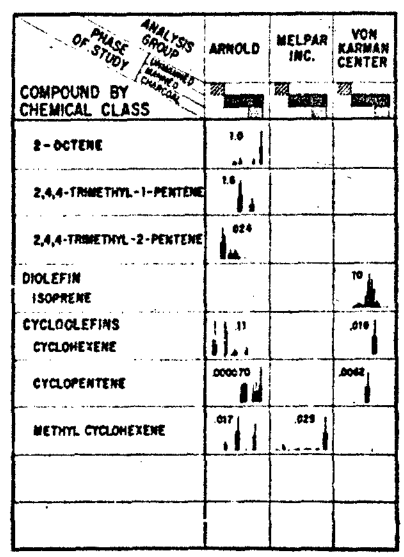

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pyuria. Urinalysis repeated 24 hours later was completely normal. Urinalyses on these subjects during the experimental phases were normal and numerous urine cultures were negative. It is noteworthy that approximately 16 hours prior to the abnormal post-experimental urinalyses, both subjects exercised moderately—their first exertion of any note since the confinement. All subjects displayed normal ability to concentrate their urine after only an 8-10 hour period of dehydration. Blood urea nitrogen, serum creatinine and endogenous creatinine clearances showed no deviations.

Subject 92 exhibited mild hyperbilirubinemia with slight elevation of the indirect fraction before the experiment. This was associated with a reticulocytosis of three percent and was probably a manifestation of a mild hemolysis or possible Gilbert's syndrome. Otherwise, liver function studies were entirely normal.

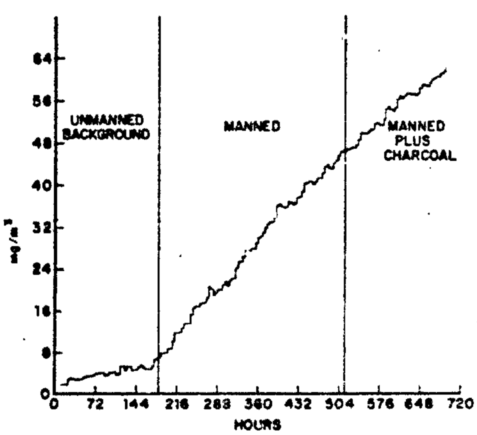

Fig. 2. Carbon monoxide concentration corrected for gas lost from the test cell. The concentrations of carbon monoxide were corrected for the amount removed from the test cell and reported as mg/m³ at 258 mm. Hg and 21.1°C.

DISCUSSION

Variance in the analysis of similarly obtained samples may result in the misinterpretation that man does or does not produce certain compounds. Many explanations may be proposed for the inconsistency of data: (1) variabilities may occur in obtaining the samples with the cryogenic trapping system, (2) condensed compounds in the trapping cylinders may alter the thermal characteristics of the cylinders, (3) interaction between molecular species could increase or decrease the efficiency of the concentration of material, (4) each analytical group developed an independent procedure for analysis, (5) each analytical group had a number of unidentifiable compounds which may have been identified by another group, (6) compounds existed in very low concentration in the test cell or supply gases, and did not reach a detectable concentration until the manned phase of the experiment, (7) compounds reported in the last phase of the experiment (manned-plus-charcoal) may have been desorbed from the activated carbon, and (8) a compound identified only by gas chromatographic elution times may be two or more compounds having the same elution time. Furthermore, variations from the absolute values of contaminants were introduced by the operation of the test cell, controlling pressure, temperature, water vapor content, and in changing the lithium hydroxide sorption canisters. Large surface areas are available within the test cell for the sorption and desorption of compounds. Man also adds a variability factor. Man's efficiency as a contaminant removal system may change, and his contaminant production may be related to physical activity and biochemical needs.

Sixty-six (66) compounds were identified during the 258 mm. Hg experiment that were not reported in the previous study conducted at 760 mm. Hg.[1] These compounds could have been present in the earlier experiment but were not reported due to the state-of-the-art of the analytical techniques at that time. As a result of

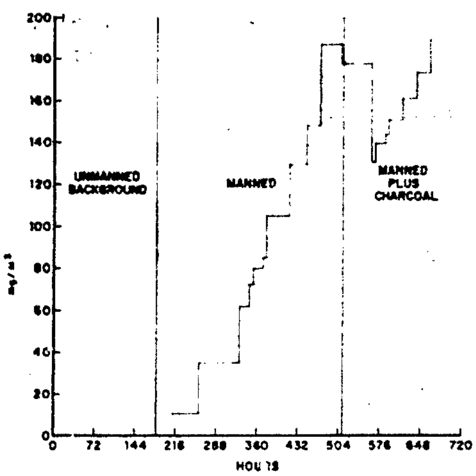

Fig. 3. Hydrogen concentration corrected for gas lost from the test cell. The hydrogen concentrations were corrected for the amount of gas removed from the test cell and reported as mg/m³ at 258 mm. Hg and 21.1°C.

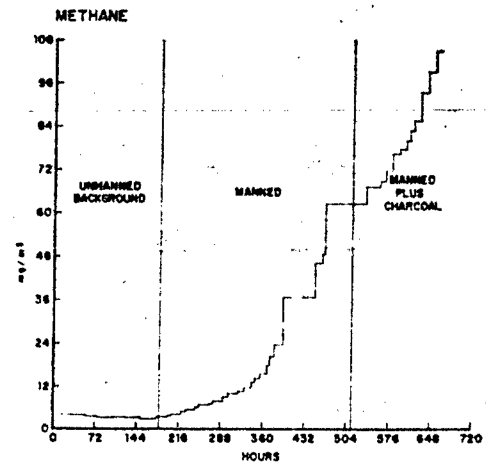

Fig. 4. Methane concentration corrected for gas lost from the test cell. The methane concentrations were corrected for the amount of methane removed from the test cell and reported as mg/m³ at 258 mm. Hg and 21.1°C.

the experience gained from the previous study, the analytical groups improved their techniques for the analysis of cryogenically obtained samples. The experience gained as a result of this experiment will enable an even more detailed identification of the components of closed-system atmospheres.

Forty-five (45) of the 142 compounds were reported only during the manned and manned-plus-charcoal phases of this experiment. Of these compounds found during both manned phases, the following 10 were detected only after the carbon scrubber had been put into operation: valeric acid, n-propyl benzene, higher amines, benzyl ether, trifluorobenzene, tetrafluorobenzene, hexafluorobenzene, dimethyl propane, methyl mercaptan, and 2-methyl-2-butene. These compounds are subject to the same limitations imposed during the manned phase with the exception of the three fluorobenzenes; the latter compounds appeared in the last two days of the experiment which suggests that they may have been associated with the activated carbon.

The data, within the limits imposed by the experimental methods, suggest that the following compounds are associated with man: hydrogen, carbon monoxide,* acetic acid,* propionic acid,* valeric acid,* methyl alcohol,* ethyl alcohol,* n-propyl alcohol, furfuryl alcohol, acetaldehyde, butyraldehyde,* furfural, mesitylene,* methyl ethyl benzene, tert-butyl benzene, diethyl benzene, sec-butyl benzene, cumene, dimethyl amine, higher amines, ethyl formate, methyl n-butyrate,* isopropyl acetate, furan,* methyl furan, benzyl ether,* tetrahydrofuran, indole, acetone,* cyclohexane, 1,2-dimethyl cyclohexane (cis), 1,2-dimethyl cyclohexane (trans), decalin,* methane,* butane, 2,3-dimethyl butane,* octane, 2,4-dimethyl pentane, decane, 2-methyl pentane, trimethyl butane, 2,3,4-trimethyl pentane, 2,2,5-trimethyl hexane, n-nonane, dimethyl sulfide, methyl mercaptan, propyl mercaptan,* methyl ethyl thiophene, thiophene, thiotolene, thioxene, ethylene, propylene, 4-methyl-2-pentene, n-heptene, 2-methyl-1-pentene, 2,4,4-trimethyl-1-pentene, hexene, 3-heptene, 1-octene, 2-octene, isoprene,* and cyclopentene.

Carbon monoxide, hydrogen, and methane increased rapidly with the inclusion of man into the test cell. The respective graphs (Figures 2, 3, 4) of the test cell concentrations, corrected for gas lost from the test cell (samples for analysis and lock operations), indicate an increase in production rate for methane and a slight decrease in the production rates of carbon monoxide and hydrogen during the manned-plus-charcoal phase of the experiment. The production rates (0°C. and 760 mm. Hg) of CO, CH_4 and H_2 during the manned phase were 0.54, 1.7, and 46.9 ml/man/hr, respectively. The values for hydrogen production are in accord with data reported by Kirk.[5]

Up to this point, the present data have been interpreted with respect to man as a source of compounds. Examples of man as a compound-removal system include the following: iso-propyl alcohol, toluene, and methyl iso-butyl ketone.

No obvious ill effects attributable to trace contaminants were discovered in the four experimental subjects who lived for 21 days in the confined environment. Minimal bends and barotalgia have been reported in previous studies of 100% oxygen at 258 mm. Hg total pressure.[4] Helvey et al.[3] noted abnormal urinary sediments from individuals living in this environment for 14 days, with the abnormality persisting for as long as 3 months. Other environmental studies by Mautner and co-workers[7] have revealed mitochondrial alterations in the proximal tubule of the monkey kidney as a result of the hyperoxic hypobaric atmosphere. In this study, the detectable but transient urinary abnormalities might possibly have been secondary to hyperoxia and less likely a result of toxicity from contaminants.

Conceivably, accumulation of trace amounts of organic compounds might have had an additive effect and be harmful to certain body systems. Minimal deviations in liver function on single determination made before and after the experiment were observed in a previous study of contaminants at ambient conditions.[1] However, detailed studies on liver function in the present work revealed no deviations during or after the environmental phase. Likewise, absence of respiratory symptoms and clear lung fields by physical examinations and chest X-ray are evidence against pulmonary difficulties. Although carbon monoxide accumulation was present, no harmful effects from this compound were detected by careful medical observations, physical examinations, and post-experimental electroencephalograms.

CONCLUSIONS

Additional information has been accumulated concerning the contaminants associated with habitation by man of a closed confined space. However, the interaction of the many contaminant removal systems (such as man, lithium hydroxide, activated charcoal, water condensation, and the walls of the test cell) modified the concentration of the contaminants preventing meaningful production rate determinations. The large volume of the test cell buffers the results, indicating the need to conduct experiments with the developed techniques and analytical procedures with a single man in a minimum volume chamber containing very few support items.

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*Compounds previously found to be suggestive of association with man in a closed system at 760 mm. Hg.[1]

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13. ABSTRACT

Additional information has been accumulated concerning the contaminants associated with habitation by man of a closed confined space. An experiment designed to determine man's contribution to trace contaminants was conducted jointly by the United States Air Force and the National Aeronautics and Space Administration. The experiment was divided into three phases: an unmanned period, a manned period, and a manned period coupled with an activated carbon scrubber. Direct analyses of the sealed environment were not adequate for this comprehensive survey; however, cryogenic fractionation and concentration provided samples with sufficient concentration of contaminants for analysis by means of gas chromatography, infrared spectroscopy, and mass spectroscopy. Of the 142 compounds identified and quantified during the experiment, only 45 were found during the manned phases.

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